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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF TIN INGOT

(First Revision)

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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July 1970

Indian Standard
METHODS OF CHEMICAL ANALYSIS OF TIN INGOT
(First Revision)

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Indian Standard
METHODS OF CHEMICAL ANALYSIS OF TIN INGOT
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 2 June 1969, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 With the publication of IS : 26-1966* and IS : 4280-1967†, need was felt to revise this standard keeping in view the impurities limits specified in these standards. The methods for determination of lead and tin in the ranges as specified in IS : 4280-1967† will be covered in a separate standard.

0.3 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960‡.

1. SCOPE

1.1 This standard prescribes the methods for determination of antimony, arsenic, copper, iron and bismuth in the ranges specified in IS : 26-1966* and IS : 4280-1967†. The method for determination of lead is suitable for analysis of lead in tin ingot. Tin in tin ingot shall be determined by difference.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with the procedure laid down in IS : 1817-1961§.

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*Specification for tin ingot (*second revision*).

†Specification for refined secondary tin.

‡Rules for rounding off numerical values (*revised*).

§Methods of sampling non-ferrous metals for chemical analysis.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1961*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF ANTIMONY BY THE POTASSIUM BROMATE METHOD

4.1 Outline of the Method — After separation of arsenic by distillation, antimony is estimated in the solution by titration with standard potassium bromate solution using methyl orange as indicator.

4.2 Reagents

4.2.1 *Potassium Bisulphate* — solid.

4.2.2 *Concentrated Sulphuric Acid* — sp gr 1·84 (conforming to IS : 266-1961†).

4.2.3 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1962‡).

4.2.4 *Sodium Chloride* — solid.

4.2.5 *Standard Potassium Bromate Solution (0·01 N)* — Dissolve 2·783 5 g of potassium bromate dried at 110°C in water and transfer the solution to 100-ml volumetric flask, washing the beaker with minimum amount of water several times. Dilute to the mark with water and mix well. Transfer 10 ml to 1-litre volumetric flask and dilute to the mark and mix well.

NOTE — For determination of antimony in refined secondary tin, 0·1 N $KBrO_3$ should be used.

4.2.6 *Methyl Orange Indicator Solution (1 g/l)* — Dissolve 0·1 g of methyl orange in 100 ml of water.

4.3 Procedure

4.3.1 Take an accurately weighed sample of 5 to 7 g in 500-ml Erlenmeyer flask. To this flask and to the second flask for blank determination, add 20 g of potassium bisulphate and 25 to 30 ml concentrated sulphuric acid. Heat slowly to decompose the sample. When decomposition is complete, heat vigorously to expel sulphur from the walls of the flask.

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*Specification for water, distilled quality (*revised*).

†Specification for sulphuric acid (*revised*).

‡Specification for hydrochloric acid (*revised*).

4.3.2 Cool to room temperature, add 15 to 20 ml of water, slowly shake and again cool to room temperature. Add 50 ml of concentrated hydrochloric acid and 10 g of sodium chloride. Connect the flask to the distillation apparatus and add 250 to 300 ml of water to the receiving beaker. Heat the contents of the distillation flask to gentle boiling, continue heating until the temperature reaches 105°C. Disconnect the flask and remove from the hot plate. Use the solution in the flask for determination of antimony and reserve the distillate for determination of arsenic. Reserve the solution.

4.3.3 To the solution reserved under **4.3.2**, add 25 g of sodium chloride. Dilute the solution with boiling water to 350 ml and titrate the solution at about 70° to 80°C with standard potassium bromate solution using methyl orange as indicator which should be added towards the end of the titration.

4.3.4 Make a blank determination following the same procedure (*see* **4.3**) and using the same amounts of all the reagents but without the sample.

4.4 Calculation

$$\text{Antimony, percent} = \frac{(A - B)\,C \times 6{\cdot}09}{D}$$

where

A = volume in millilitres of the standard potassium bromate solution used for titration of the sample,

B = volume in millilitres of the standard potassium bromate solution required for blank determination,

C = normality of the standard potassium bromate solution, and

D = weight in grams of the sample used.

5. DETERMINATION OF ARSENIC BY THE MOLYBDENUM BLUE (PHOTOMETRIC) METHOD

5.1 Outline of the Method — Arsenic is separated as arsenious chloride by distillation and determined photometrically by molybdenum blue method. The measurement is made at about 660 nm (nanometre).

5.2 Reagents

5.2.1 *Methyl Orange Solution (1 g/l)* — Dissolve 0·1 g of methyl orange in 100 ml water.

5.2.2 *Dilute Ammonium Hydroxide* — 1 : 1 (*v/v*).

5.2.3 *Dilute Hydrochloric Acid* — 1 : 9 (*v/v*).

5.2.4 *Potassium Bromate Solution* — 0·3 g/l.

5.2.5 *Ammonium Molybdate Solution (5 g/l)* — Transfer 0·5 g of ammonium molybdate [$(NH_4)_6Mo_7O_{24}, 4H_2O$] to 100-ml volumetric flask containing 40 ml water. Shake to dissolve the salt. Dilute to the mark and mix well. Use the freshly prepared solution.

5.2.6 *Hydrazine Sulphate Solution (1 g/l)* — Dissolve 0·1 g of hydrazine sulphate in water. Take the solution in 100-ml measuring flask and dilute to the mark. Use the freshly prepared solution.

5.2.7 *Standard Arsenic Solution (1 ml = 0·01 mg* As) — Take 0·132 0 g of standard arsenic As_2O_3 in 1-litre measuring flask. Add 10 ml of concentrated hydrochloric acid and dissolve the oxide. Dilute to the mark with water and mix well. Take 100 ml of the solution to 1-litre measuring flask. Add 40 ml of concentrated hydrochloric acid and dilute to the mark. Mix well.

5.3 Procedure

5.3.1 Transfer the distillate reserved under **4.3.2** to 500-ml measuring flask, dilute to the mark and mix well. Then transfer an aliquot containing 0·01 to 0·2 mg arsenic to 50-ml measuring flask. To this, add 2 drops of methyl orange solution and then neutralize with dilute ammonium hydroxide till yellow colour of the indicator is developed. Add drop by drop dilute hydrochloric acid till the colour changes to red. Add 10 ml of dilute hydrochloric acid and 1 ml of the potassium bromate solution. Heat the flask on a hot plate maintained nearly at 50°C when methyl orange and arsenic will be oxidized. To the warm solution, add 5 ml of ammonium molybdate solution and shake to mix well. Add 2·0 ml of hydrazine sulphate solution, dilute to the mark in warm condition and mix well. Place the flask in a water-bath filled with boiling water. Keep the flask for 10 minutes. Remove the flask, shake well and cool to room temperature.

5.3.2 Transfer a suitable portion of the solution to the absorption cell and take photometric reading using a light band centred approximately at 660 nm.

5.3.3 *Calibration Curve* — Transfer 1 ml to 16 ml of standard arsenic solution to 50-ml volumetric flasks. Using the same quantities of reagents, carry out the entire stages of procedures as described under **5.3.1** and **5.3.2**, and record the photometric readings of all the standard solutions against the blank.

5.4 Calculation — Convert the photometric reading of the sample to milligrams of arsenic by means of a calibration curve and calculate the percentage of arsenic as follows:

$$\text{Arsenic, percent} = 0{\cdot}1 \times \frac{A}{B}$$

where

A = weight in milligrams of arsenic found in the aliquot, and

B = weight in grams of the sample represented by the aliquot taken.

6. DETERMINATION OF COPPER BY THE CUPRIC BROMIDE (PHOTOMETRIC) METHOD

6.1 Outline of the Method — Cupric copper forms a red-violet coloured complex in hydrobromic acid. Photometric measurement is made at about 600 nm.

6.2 Reagents

6.2.1 *Hydrobromic Acid-Bromine Mixture* — Add 10 ml bromine to 90 ml of hydrobromic acid.

6.2.2 *Hydrobromic Acid* — 48 percent.

6.2.3 *Bromine Solution* — saturated.

6.2.4 *Concentrated Nitric Acid* — sp gr 1·42 (conforming to IS : 264-1968*).

6.2.5 *Perchloric Acid* — 70 percent.

6.2.6 *Test Lead* — finely granulated test lead which contains less than 0·000 1 percent copper and less than 0·001 percent iron and nickel.

6.2.7 *Phosphoric Acid* — 85 percent (*w/v*).

6.2.8 *Standard Copper Solution* (*1 ml = 0·1 mg* Cu) — Dissolve 0·1 g of copper in 3 ml of concentrated nitric acid by gently heating in 100-ml conical flask. Add 10 ml of perchloric acid and continue heating till the copious fumes of perchloric acid are formed and nitric acid is expelled completely. Cool, add 20 ml of water and pour into 1-litre volumetric flask and dilute to the mark.

6.3 Procedure

6.3.1 Weigh accurately 1 g of sample in 150-ml beaker. Add 20 ml of hydrobromic acid-bromine mixture. Heat gently avoiding excessive loss of bromine, thereby helping the dissolution of the sample. Add a few drops of bromine more for complete dissolution of the sample and conversion of the tin to the stannic form. Heat the solution to dryness and add 10 ml of hydrobromic acid-bromine mixture to the beaker. Heat till the residue dissolves. Continue heating so that major amount of tin and almost all antimony is expelled. Boil gently to remove the bromine completely and evaporate the solution to dryness.

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*Specification for nitric acid (*first revision*).

6.3.2 Add 3 ml of concentrated nitric acid and boil to expel the trace of bromine. Add 10 ml of perchloric acid and boil till the white fumes are given off. Dilute the solution to 50 ml. Add 1 g of test lead and boil for 10 minutes at moderate heat till all copper is precipitated. Cool, allow to settle and decant through pulp pad; wash the pulp with water. Reserve the filtrate for iron determination.

6.3.3 Dissolve the copper on the pulp pad with concentrated nitric acid from a dropping bottle and collect the solution in the original beaker containing copper and lead. Wash the pad with minimum amount of water. Heat the solution to dryness.

6.3.4 Add 10 ml of hydrobromic acid-bromine mixture and heat gently to dissolve the residue. When the dissolution is complete, boil the solution to remove excess bromine. Cool to room temperature.

6.3.5 Transfer the solution to 25-ml volumetric flask containing 10 ml of phosphoric acid. Wash the sides of the flask with a few millilitres of hydrobromic acid. Dilute to the mark with hydrobromic acid and mix well.

6.3.6 Transfer a suitable portion of the solution to the absorption cell and take photometric reading using a light band centred approximately at 600 nm.

6.3.7 *Calibration Curve* — Transfer 1 ml to 20 ml of the standard copper solution to 25-ml volumetric flasks. Using the same quantities of reagents, carry out the entire stages of procedures as described under **6.3.5** and **6.3.6**, and record the photometric readings of all the standard solutions against the blank.

6.4 Calculation — Convert the photometric reading of the sample to milligrams of copper by means of a calibration curve and calculate the percentage of copper as follows:

$$\text{Copper, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in milligrams of copper present in the solution, and

B = weight in grams of the sample represented by the aliquot.

7. DETERMINATION OF IRON BY THE ORTHO-PHENANTHROLINE (PHOTOMETRIC) METHOD

7.1 Outline of the Method — Ferrous iron forms orange-red complex with *o*-phenanthroline at *p*H 4. Measurement is made at about 510 nm.

7.2 Reagents

7.2.1 *Sodium Acetate Acetic Acid Buffer Solution* — Dissolve 270 g of anhydrous sodium acetate in 500 ml of water, add 240 ml of acetic acid and dilute to 1 litre.

7.2.2 *Hydroxylamine Hydrochloride Solution* (*10 g/l*) — Dissolve 1 g of the reagent in water. Transfer the solution to 100-ml volumetric flask. Dilute to the mark and mix well.

7.2.3 *Ortho-Phenanthroline Solution* (*2 g/l*) — Dissolve 0·2 g of *o*-phenanthroline monohydrate in 90 ml of water with gentle heating and constant stirring. Cool and dilute to 100 ml.

7.2.4 *Standard Iron Solution* (*1 ml = 0·02 mg* Fe) — Dissolve 0·2 g of pure iron in concentrated hydrochloric acid. Dilute the solution to 100 ml. Take the solution in 1-litre volumetric flask, dilute to the mark and mix well. Take 100 ml of this solution to 1-litre measuring flask and dilute to the mark with water. Mix well.

7.3 Procedure

7.3.1 Transfer the solution reserved under **6.3.2** to 100-ml volumetric flask, washing the beaker with water. Dilute to the mark. Transfer an aliquot to 100-ml volumetric flask. Add 10 ml of buffer solution, 2·0 ml of hydroxylamine hydrochloride solution and mix well. Add 5 ml of *ortho*-phenanthroline solution, dilute to the mark and shake for proper mixing.

7.3.2 Transfer a suitable portion of the solution to the absorption cell and take photometric reading using a light band centred approximately at 490 nm.

7.3.3 *Calibration Curve* — Transfer 1 ml to 16 ml of the standard iron solution in 100-ml volumetric flasks, using same quantities of reagents, carry out the entire stages of procedures as described under **7.3.1** and **7.3.2** and record the photometric readings of all the standard solutions against the blank.

7.4 Calculation — Convert the photometric reading of the sample to milligrams of iron by means of a calibration curve and calculate the percentage of iron as follows:

$$\text{Iron, percent} = 0{\cdot}1 \times \frac{A}{B}$$

where

A = weight in milligrams of iron found in aliquot, and

B = weight in grams of the sample represented by the aliquot.

8. DETERMINATION OF BISMUTH BY THE THIOUREA (PHOTOMETRIC) METHOD

8.1 Outline of the Method — Bismuth forms with thiourea a yellow coloured complex which is measured photometrically at about 440 nm.

8.2 Reagents

8.2.1 *Hydrobromic Acid-Bromine Mixture* — Mix 20 ml of bromine and 180 ml of hydrobromic acid.

8.2.2 *Bromine Solution* — saturated.

8.2.3 *Concentrated Nitric Acid* — *See* **6.2.4**.

8.2.4 *Perchloric-Phosphoric Acid Mixture* — Mix 160 ml of phosphoric acid with 840 ml of perchloric acid.

8.2.5 *Fluoboric Acid Solution* — Mix 45 ml of saturated solution of boric acid and 5 ml of hydrofluoric acid and dilute the mixture to 500 ml.

8.2.6 *Thiourea Solution (80 g/l)* — Dissolve 20 g of thiourea in 150 ml cold water. Transfer the solution to 250-ml volumetric flask, washing the sides of the beaker with minimum amount of water. Dilute to the mark and mix well.

8.2.7 *Standard Bismuth Solution (1 ml = 0·1 mg* Bi) — Take 0·1 g of pure bismuth metal in 150-ml beaker. Dissolve the metal in 15 ml of dilute nitric acid (1 : 3). Heat gently when brown fumes will be given off. Cool and transfer the solution to 1-litre volumetric flask. Dilute to the mark with dilute nitric acid (1 : 9).

8.3 Procedure

8.3.1 Take 1 g of an accurately weighed sample in 250-ml wide-mouth flask. Add 20 ml of hydrobromic acid-bromine mixture. Heat gently for the complete dissolution of the sample. Add few drops of bromine solution for better dissolution of the sample. Heat to dryness and repeat the operation several times in order to expel most of arsenic, antimony and tin.

8.3.2 Add few drops of concentrated nitric acid and boil to decompose the bromides. Then add 12 ml of perchloric-phosphoric acid mixture and continue heating till the white copious fumes of perchloric acid are given off.

8.3.3 Transfer the solution to 100-ml volumetric flask and add 5 ml of fluoboric acid solution and mix well. Add 25 ml of freshly prepared thiourea solution. Transfer the whole solution to 200-ml volumetric flask, cool to room temperature and dilute to the mark. Mix well. Transfer a suitable portion of solution to the absorption cell and take photometric reading using a light band centred approximately at 440 nm.

8.3.4 *Calibration Curve* — Transfer 1 to 25 ml of standard bismuth solutions to 100-ml volumetric flasks. Using the same quantities of reagents, carry out the entire stages of procedures as described under **8.3.3** and record the photometric readings of all the standard solutions against the blank.

8.4 Calculation — Convert the photometric reading of the sample to milligrams of bismuth by means of a calibration curve and calculate the percentage of bismuth as follows:

$$\text{Bismuth, percent} = 0{\cdot}1 \times \frac{A}{B}$$

where

A = weight in milligrams of bismuth found, and

B = weight in grams of the sample represented by the aliquot.

9. DETERMINATION OF LEAD BY THE DITHIZONE (PHOTOMETRIC) METHOD (FOR LEAD CONTENT UP TO 0·1 PERCENT)

9.1 Outline of the Method — Lead dithizonate, formed in buffered cyanide solution, is extracted with chloroform solution of dithizone. The excess dithizone in the chloroform is removed by ammoniacal sodium sulphite solution of higher *p*H. Photometric measurement of lead dithzonate is made at about 515 nm.

9.2 Reagents

9.2.1 *Hydrobromic Acid-Bromine Mixture* — *See* **6.2.1**.

9.2.2 *Bromine Solution* — saturated.

9.2.3 *Concentrated Nitric Acid* — *See* **6.2.4**.

9.2.4 *Dilute Nitric Acid* — 2 : 98 (*v*/*v*).

9.2.5 *Dilute Ammonium Hydroxide* — 1 : 1 (*v*/*v*).

9.2.6 *Hydrogen Peroxide* — 20 volumes.

9.2.7 *Buffer Solution* (*A*) — Dissolve 100 g of sodium citrate and 100 g of ammonium acetate in water and dilute to 500 ml.

9.2.8 *Buffer Solution* (*B*) — In 1-litre volumetric flask, dissolve 1 g of sodium sulphite in 300 ml of water. Add 475 ml of dilute ammonia (1 : 1) and 20 ml of potassium cyanide solution. Dilute to the mark with water and mix well.

9.2.9 *Hydroxylamine Hydrochloride* — solid.

9.2.10 *Dilute Acetic Acid* — 1 : 4 (*v*/*v*).

9.2.11 *Potassium Cyanide Solution* (*200 g/l*) — Dissolve 200 g of potassium cyanide in water and dilute to 1 litre. It should be free from lead and sulphides and undissolved suspended material. If the solution is turbid, filter through a hardened paper and store the solution in a polyethylene bottle.

CAUTION — The preparation, storage, and use of potassium cyanide solution require care and attention. Avoid inhalation of fumes and exposure of skin to the chemical or its solution. Work in a well-ventilated room.

9.2.12 *Dithizone Solution* — Dissolve 0·025 g of the reagent in chloroform; dilute to 250 ml with chloroform and mix well.

9.2.13 *Dilute Dithizone Solution* — Dilute 25 ml of the stock solution (*see* **9.2.12**) to 250 ml with chloroform and mix. Prepare the solution just before use.

9.2.14 *Standard Lead Solution* (*1 ml = 0·001 mg* Pb) — Dissolve 0·1 g of lead in 20 ml of dilute nitric acid (1 : 1), heat to dissolve the metal and to expel the brown fumes. Cool, transfer the solution to 1-litre volumetric flask, washing the sides of the beaker several times with water. Dilute to the mark and mix well. Dilute 5 ml to 500 ml in a volumetric flask and mix well. Use freshly prepared solution.

9.3 Procedure

9.3.1 Take 1 to 2 g of an accurately weighed sample in 125-ml beaker. Add 40 ml of hydrobromic acid-bromine mixture. Heat at moderate temperature so that bromine fumes are not expelled completely. Add few drops of bromine solution for complete dissolution of the sample. Heat slowly to dryness at moderate temperature. Repeat the process in order to expel major amount of tin.

9.3.2 Add 10 ml of concentrated nitric acid to the dried mass and boil to remove bromine completely. Add 15 to 20 ml of hot water and continue boiling when traces of tin, if present, will be precipitated. Filter through pulp pad and wash the precipitate with dilute nitric acid. Reject the residue.

9.3.3 Neutralize filtrate with dilute ammonium hydroxide and maintain acidity of the filtrate to about 3 percent of nitric acid. Electrolyze the solution using current density of 4 A/dm^2. When the deposition is complete, dissolve the lead deposit on the anode in mixture of concentrated nitric acid and hydrogen peroxide. Boil to remove hydrogen peroxide and transfer the solution to 100-ml volumetric flask. Dilute to the mark and mix well.

9.3.4 Transfer an aliquot to 100-ml conical flask and add 5·0 ml buffer solution (*A*) and 10 mg of hydroxylamine hydrochloride. Add dilute ammonium hydroxide slowly, and adjust the *p*H of solution with the help of universal indicator paper to 8. Add 10 ml of dilute acetic acid and cool. Transfer the solution to 250-ml separatory funnel, washing the flask with minimum amount of water. Add 20 ml of potassium cyanide solution to the funnel. Add 25 ml of dilute dithizone solution to the funnel stopper and shake vigorously. Allow to stand for 3 minutes to separate the layers. Transfer the chloroform layer to the second separatory funnel. Add 50 ml of buffer solution (*B*). The bore of the stem of the second funnel shall be dry. Shake vigorously for 2 minutes. Allow to stand to separate the layers. Draw off a few millilitres of chloroform layer and discard. Filter a suitable portion of chloroform layer through the filter paper into an absorption cell and take the photometric readings using a light band centred approximately at 575 nm.

9.3.5 *Calibration Curve* — Transfer 5 to 35 ml of standard lead solutions to 250-ml separatory funnels. Using the same quantities of reagents, carry out the entire stages of procedures as described under **9.3.4** and record the photometric readings of all the standard solutions against the blank.

9.4 Calculation — Convert the photometric reading of the sample solution to milligrams of lead by means of a calibration curve and calculate the percentage of lead as follows:

$$\text{Lead, percent} = 0{\cdot}1 \times \frac{A}{B}$$

where

A = weight in milligrams of lead found, and

B = weight in grams of the sample represented by the aliquot.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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5-8-56C L. N. Gupta Marg	HYDERABAD 500001	22 10 83
R 14 Yudhister Marg, C Scheme	JAIPUR 302005	6 98 32
117/418 B Sarvodaya Nagar	KANPUR 208005	4 72 92
Patliputra Industrial Estate	PATNA 800013	6 28 08
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27

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